

## MISSOURI DEPARTMENT OF TRANSPORTATION MATERIALS ENGINEERING Jefferson City, Missouri

# Test Method MoDOT T58 DETERMINATION OF PHOSPHORIC ACID (P2 O5) IN COMMERCIAL FERTILIZERS

- **1.0 Scope.** This method describes a procedure for determining the percent phosphoric acid in commercial fertilizers by colorimetry.
- 2.0 Reagents and Apparatus.
- **2.1** An Atomic Absorption Spectrophotometer or a suitable colorimeter.
- 2.2 Nitric Acid (HNO<sub>3</sub>), 1.42 specific gravity.
- **2.3** Hydrochloric Acid (HCl), 1.19 specific gravity.
- **2.4** Potassium Phosphate, Monobasic (KH<sub>2</sub>PO<sub>4</sub>), Primary Standard Grade, dried at 105-110C for several hours prior to use.
- 2.5 Ammonium Molybdate ((NH<sub>4</sub>)<sub>6</sub>MO<sub>7</sub>O<sub>2</sub>4•4H<sub>2</sub>O), Reagent Grade.
- **2.6** p-Methylaminophenol Sulfate, Reagent Grade.
- 2.7 Sodium Acid Sulfite (NaHSO<sub>3</sub>), Reagent Grade.
- **2.8** Cuvets, optical glass, matched, 10mm path length. (Note: If available, an optical glass, automatic flow-through cuvet may be used.)
- 3.0 Preparation of Standard Solutions.
- 3.1 Phosphate Stock Solution: Weigh 0.4394 g of dried, primary standard  $KH_2PO_4$  into a 500-mL volumetric flask. Add 30 mL of HNO<sub>3</sub> and 5 mL of HCl and boil until brown fumes have been expelled. Dilute to volume with distilled water.
- 5 ppm Working Standard: Pipette a 5-mL aliquot of the phosphate stock solution into a 200-mL volumetric flask and dilute to volume with distilled water. This solution is equivalent to 57.2% phosphoric acid.



TM-58 (Page 2 of 3) (Rev. 05-01-00)

- 3.3 4 ppm Working Standard: Pipette a 4-mL aliquot of the phosphate stock solution into a 200-mL volumetric flask and dilute to volume with distilled water. This solution is equivalent to 45.8% phosphoric acid.
- 3.4 Blank Working Solution: Boil a mixture consisting of 5 mL of HCl and 30 mL of HNO<sub>3</sub> until brown fumes have been expelled, transfer to a 500-mL volumetric flask and dilute to volume with distilled water. Transfer a 4-mL aliquot to a 200-mL volumetric flask and dilute to volume with distilled water.

## 4.0 Preparation of Color Development Reagents.

- **4.1** Acid Molybdate Solution: Pour, with stirring, a solution containing 16.62 g of ammonium molybdate in 156.9 mL of distilled water into a solution of 318.4 mL of HCl to which 29.6 mL of distilled water has been added.
- **4.2** Reducing Solution: Dissolve 1.0 g of p-methylaminophenol sulfate and 3.0 g of sodium acid sulfite in 100 mL distilled water.

### 5.0 Procedure.

- 5.1 Weigh, to the nearest 0.1 mg,  $0.50 \pm 0.02$  g of sample into a 500-mL volumetric flask, adding 30 mL of HNO3 and 5 mL of HCl. Boil until brown fumes have been expelled, cool to room temperature and dilute to volume with distilled water. Transfer a 2mL aliquot to a 100-mL volumetric flask and dilute to volume with distilled water. From this second dilution, transfer a 10-mL aliquot to a 20-mL scintillation vial and add a 2-mL aliquot of acid molybdate solution and a 2-mL aliquot of reducing solution. Allow to stand 1 hour for color development. At the same time that the sample vial is prepared, a blank vial, a 4 ppm phosphorus vial and a 5 ppm phosphorus vial must also be prepared and allowed to develop color along with the sample. The blank, 4 ppm phosphorus and 5 ppm phosphorus are prepared by the same procedure used for preparation of the sample, using 10-mL aliquots of the blank working solution, the 4 ppm phosphorus working standard and the 5 ppm phosphorus working standard. (Note: When making determinations on fertilizers with low percentages of phosphoric acid, it is necessary to adjust the sample weight so that the sample working dilution contains between 4 ppm and 5 ppm phosphorus.)
- 5.2 Calibrate the instrument using the blank solution, the 4 ppm phosphorus solution and the 5 ppm phosphorus solution, then determine the concentration of the sample solution. (Note: When using an Atomic Absorption Spectrophotometer for color measurements, the cuvet holder is placed over the burner in the light path and a Cu hollow cathode lamp is used with the instrument grating set at a wavelength of 640 nm.)

#### **6.0** Calculations.

**6.1** The method of calculating the % phosphoric acid will vary according to the make and model of instrument used.



Report the results to the nearest 0.1%  $P_2O_5$  as follows:

 $\% \ Phosphoric \ Acid \ (P_2O_5)$ 

